REMARKS/ARGUMENTS

Reconsideration of this application is requested. Claims 19-43 are in the case. It is noted with appreciation that claims 19-36 are free of the art and will be allowable once the formal matters are resolved.

I. DECLARATION

An executed substitute declaration showing the serial number and filing date is attached hereto. Entry of the declaration into the record of this case is respectfully requested.

II. THE 35 U.S.C. §112, SECOND PARAGRAPH, REJECTION

Claims 19-36 stand rejected under 35 U.S.C. §112, second paragraph, as allegedly indefinite for the reasons stated on page 2 of the Action. In response, and without conceding to the merit of this rejection, claims 19 and 30 have been amended to recite that the HTHP fluid loss is determined using an HTHP test according to the specification of the American Petroleum Institute (API) that employs a pressurized cell fitted with a filtration medium comprising a standard hardened filter paper having a filtration area of 3.5 square inches (2.258×10^{-3} m^2) wherein the drilling mud is filtered using the cell at a temperature corresponding to the temperature in the wellbore and with a standard pressure differential across the filter paper of 500 psi (3.45 MPa) and wherein a filter cake is allowed to build up on the filter paper for a period of 30 minutes, and the volume of filtrate collected after this 30 minute period is doubled to give a corrected American Petroleum Institute (API) fluid loss value. Support for this

amendment appears at page 4, line 24 to page 5, line 8 of the originally filed application. For completeness, copies of the respective Recommended Practice Standard Procedure for Field Testing Water-Based Drilling Fluids 13B-1 and 13B-2 are attached. No new matter is entered.

Two new claims are presented, the first dependent upon claim 19 and the second dependent upon claim 30, claiming the feature that the "test temperature is in the range 50 to 150°C. Support for these new dependent claims is provided at page 5, line 9. No new matter is entered.

Favorable action is awaited.

Respectfully submitted,

NIXON & VANDERHYE P.C.

By: _____
Leonard C. Mitchard
Reg. No.

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Attachments: Executed substitute declaration; Recommended Practice Standard Procedures for Field Testing Water-Based Drilling Fluids 13B-1 and 13B-2.

Recommended Practice Standard Procedure for Field Testing Water-Based Drilling Fluids

API RECOMMENDED PRACTICE 13B-1
SECOND EDITION, SEPTEMBER 1997

American Petroleum Institute

e. Stir the drilling fluid sample for 10 seconds at high speed.
f. Allow the drilling fluid sample to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a 3-revolutions per minute speed, the maximum reading attained after starting rotation at 3 revolutions per minute is the initial gel strength. Record the initial gel strength (10 sec gel) in pounds per 100 square feet (pascals).
g. Restir the drilling fluid sample at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in 4.3.2, Item f, and report the maximum reading as 10-minute gel in pounds per 100 square feet (pascals).

4.3.3 Calculation

The following calculation should be used:

$$\textit{Plastic Viscosity, cP} = \begin{bmatrix}\textit{600 rpm}\\ \textit{reading}\end{bmatrix} - \begin{bmatrix}\textit{300 rpm}\\ \textit{reading}\end{bmatrix}$$

$$\textit{Yield Point, lb/100 ft}^2 = \begin{bmatrix}\textit{300 rpm}\\ \textit{reading}\end{bmatrix} - \textit{Plastic Viscosity}$$

$$\textit{Apparent Viscosity, cP} = \frac{\textit{600 rpm reading}}{2}$$

5 Filtration

5.1 DESCRIPTION

Measurement of the filtration behavior and wall-cakebuilding characteristics of a mud are fundamental to drilling-fluid control and treatment, as are the characteristics of the filtrate, such as oil, water, or emulsion content.

These characteristics are affected by the types and quantities of solids in the fluid and their physical and chemical interactions, which, in turn, are affected by temperature and pressure. Therefore, tests are run at both low pressure/low temperature and high pressure/high temperature, and each requires different equipment and techniques.

5.2 LOW-TEMPERATURE/LOW-PRESSURE TEST

5.2.1 Equipment—Low-Temperature/Low-Pressure Test

The following equipment is needed for the low-temperature/low-pressure test:

a. A filter press: This consists mainly of a cylindrical mud cell having an inside diameter of 3 inches (76.2 millimeters) and a height of at least 2.5 inches (64.0 millimeters). This chamber is made of materials resistant to strongly alkaline solutions and is so fitted that a pressure medium can be conveniently admitted into, and bled from, the top. The arrangement is also such that a sheet of 9-centimeter filter paper can be placed in the bottom of the chamber just above a suitable support. The filtration area is 7.1 ±0.1 cubic inches (4580 ±60 cubic millimeters). Below the support is a drain tube for discharging the filtrate into a graduated cylinder.

Sealing is accomplished with gaskets, and the entire assembly supported by a stand. Figures 10 and 11 depict standard units by different manufacturers.

Pressure can be applied with any nonhazardous fluid medium, either gas or liquid. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midget pressure cartridges, or means for utilizing hydraulic pressure.

To obtain correlative results, one thickness of the proper 90-millimeter filter paper, Whatman No. 50, S&S No. 576, or the equivalent, must be used.

Note: The mini-press or half-area press does not directly correlate with the results obtained when using the standard-sized press.

Note: The API low-temperature/low-pressure filter press must have a filter area of 4520 square millimeters to 4640 square millimeters, which is a diameter of 75.86 millimeters to 76.86 millimeters. The filter press gasket is the determining factor of the filter area. It is recommended that a filter press gasket used be tested by a conical gauge that has the maximum (76.86 millimeters) and the minimum (75.86 millimeters) marked on it. Any filter press gasket found out of these ranges (either larger or smaller than the markings) will be discarded.

b. Timer: 30-minute interval.
c. Graduated cylinder (TC): 10-cubic centimeter or 25-cubic centimeter.

5.2.2 Procedure—Low-Temperature/Low-Pressure Test

This procedure should be followed for the low-temperature/low-pressure test:

a. Be sure each part of the cell, particularly the screen, is clean and dry, and that the gaskets are not distorted or worn. Pour the mud sample into the cell to within 0.5 inch (13 millimeters) to the top (to minimize CO_2 contamination of filtrate) and complete the assembly with the filter paper in place.
b. Place a dry graduated cylinder under the drain tube to receive the filtrate. Close the relief valve and adjust the regulator so that a pressure of 100 ±5 pounds per square inch (690 ±35 kilopascals) is applied in 30 seconds or less. The test period begins at the time of pressure application.
c. At the end of 30 minutes, measure the volume of filtrate. Shut off the flow through the pressure regulator and open the relief valve carefully. Report the time interval, if other than 30 minutes.
d. Report the volume of filtrate in cubic centimeters (to 0.1 cubic centimeter) as the API filtrate, and the initial mud temperature in °F (°C). Save the filtrate for appropriate chemical testing.

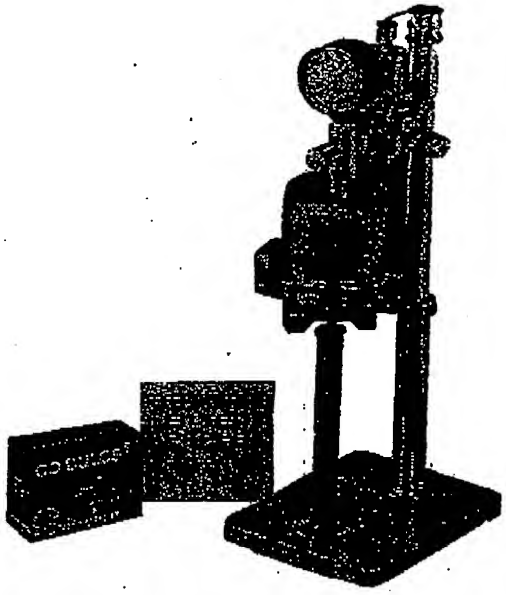
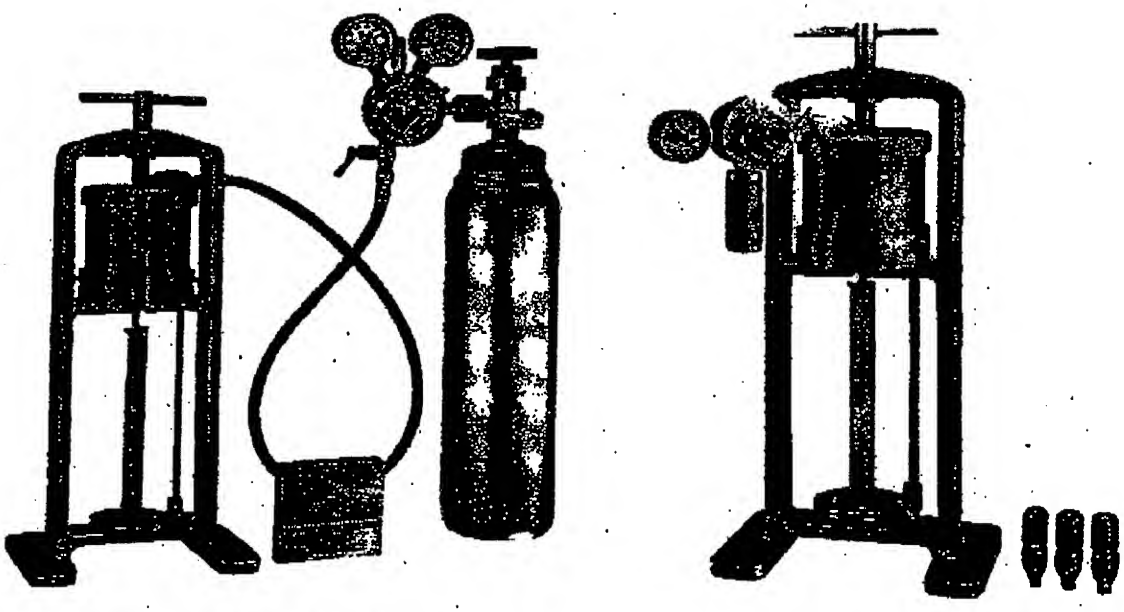


Figure 10—Filter Press With CO_2 Cartridge Pressurization



WITH NITROGEN PRESSURIZATION

WITH CO_2 CARTRIDGE PRESSURIZATION

Figure 11—Filter Press

e. Remove the cell from the frame, first making certain that all pressure has been relieved. Using extreme care to save the filter paper with a minimum of disturbance to the cake, disassemble the cell and discard the mud. Wash the filter cake on the paper with a gentle stream of water.
f. Measure and report the thickness of the filter cake to the nearest 1/32 inch (0.8 millimeter).
g. Although cake descriptions are subjective, one may use such notations as *hard, soft, tough, rubbery, firm,* and the like, to convey important information of cake quality.

5.3 HIGH-TEMPERATURE/HIGH-PRESSURE TEST

5.3.1 Equipment—High-Temperature/High-Pressure Test

CAUTION: All manufacturers' equipment are not capable of the same temperatures and pressures. Rigid adherence to manufacturers' recommendations as to sample volumes, temperatures, and pressures is essential. Failure to do so could result in serious injury.

The following equipment is used for the high-temperature/high-pressure test:

a. The high-temperature/high-pressure filter press: This consists of a controlled pressure source (CO_2 or nitrogen), regulators, a mud cell able to contain working pressures from 600 pounds per square inch to 1300 pounds per square inch, a system for heating the cell, a pressurized collection cell able to maintain proper back pressure (see Table 2) in order to prevent flashing or evaporation of the filtrate, and a suitable stand. Figures 12 and 13 show available units. The mud cell has a thermometer well, oil-resistant gaskets, a support for the filter medium, and a valve on the filtrate delivery tube to control flow from the cell. It may be necessary to replace the gaskets frequently.

CAUTION: Nitrous oxide cartridges should not be used as pressure sources for high-temperature, high-pressure (HTHP) filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil, or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

b. Filter medium:
   1. Whatman No. 50 or equivalent filter paper for temperatures to 400°F (204°C).
   2. Dynalloy X-5 or equivalent porous disc for temperatures above 400°F (204°C). A new disc is required for each test.
c. Timer: 30-minute interval.
d. Thermometer: up to 500°F (260°C).
e. Graduated cylinder (TC): 25-cubic centimeter or 50-cubic centimeter.
f. High-speed mixer.

5.3.2 Procedure—Temperature to 300°F (149°C)

Follow this procedure for the high-temperature/high-pressure test to 300°F (149°C).

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain desired temperature.
b. Stir the mud sample for 10 minutes with a high-speed mixer. Pour the mud sample into the mud cell, being careful not to fill closer than 0.5 inch (13 millimeters) from the top to allow for expansion. Install the filter paper.
c. Complete the assembly of the cell and, with both top and bottom valves closed, place it in the heating jacket. Transfer the thermometer to the well in the mud cell.
d. Connect the high-pressure collection cell to the bottom valve and lock in place.
e. Connect a regulated pressure source to the top valve and collection cell, and lock in place.
f. Keeping the valves closed, adjust the top and bottom regulators to 100 pounds per square inch (690 kilopascals). Open the top valve, applying 100 pounds per square inch (690 kilopascals) to the mud. Maintain this pressure until the desired temperature is stabilized. The time of heating of the sample in the filter cell should never exceed a total of one hour.
g. When the sample reaches the selected temperature, increase the pressure of the top pressure unit to 600 pounds per square inch (4140 kilopascals) and open the bottom valve to start filtration. Collect the filtrate for 30 minutes, maintaining the selected temperature within ±5°F (±3°C). If back pressure rises above 100 pounds per square inch (690 kilopascals) during the test, cautiously reduce the pressure by drawing off a portion of the filtrate. Record the total volume, temperature, pressure, and time.
h. Correct the filtrate volume to a filter area of 7.1 square inches (4580 square millimeters). If the filter area is 3.5 square inches (2258 square millimeters), double the filtrate volume, and report that volume.
i. At the end of the test, close the top and bottom valves on the mud cell. Bleed the pressure from the regulators.

CAUTION: Pressure in the mud cell will still be approximately 500 pounds per square inch (3450 kilopascals). Keep the cell upright and cool it to room temperature before disassembling. Bleed the pressure from the cell before disassembling.

j. Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off the regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from the cell contents, and open the cell. Discard the mud, and retrieve the filter cake. Wash the filter cake on the paper with a gentle stream of water.
k. Measure and report the thickness of the filter cake to the nearest 1/32 inch (0.8 millimeter).

Table 2—Recommended Minimum Back Pressure

Test Temperature		Vapor Pressure		Minimum Back Pressure	
°F	°C	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
		LIMIT of "Normal" Field Testing			
[a]350	177	135	932	160	1104
[a]400	204	247	1704	275	1898
[a]450	232	422	2912	450	3105

[a]Do not exceed equipment manufacturers' recommendations for maximum temperatures, pressures, and volumes.

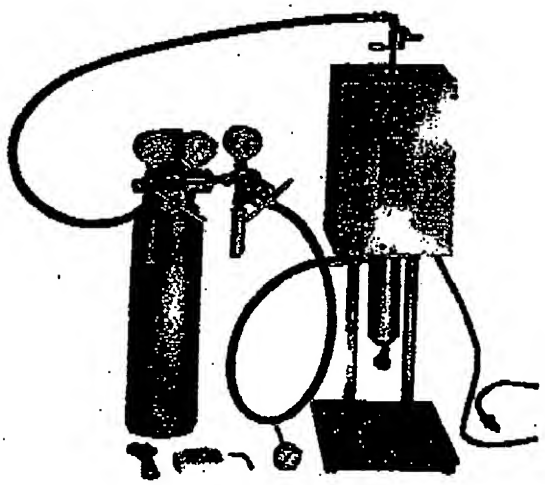


Figure 12—High-Temperature Filter Press, I

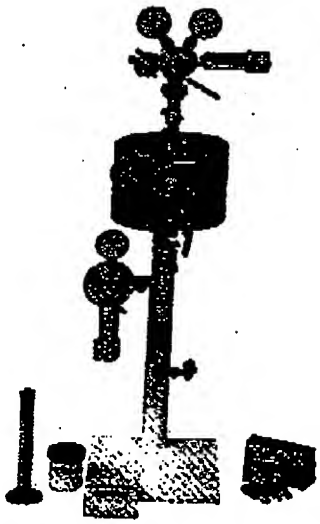


Figure 13—High-Temperature Filter Press, II

5.3.3 Procedure—Temperature Above 300°F (149°C).

CAUTION: Not all the manufacturers' equipment can be used above 300°F (149°C). Know the pressure/temperature rating of equipment in use. Failure to do so could result in serious injury.

Testing at high temperature and high pressure calls for added safety precautions. All pressure cells should be equipped with manual relief valves. Heating jackets should be equipped with both an overheat safety fuse and thermostatic cutoff. Vapor pressure of the liquid phase of muds becomes an increasingly critical design factor as test temperatures are raised. Water vapor pressures at various temperatures are shown in Table 2.

Follow this procedure for high-temperature/high-pressure tests above 300°F (149°C):

a. Place the thermometer in the well in the jacket and preheat to 10°F (6°C) above the desired temperature. Adjust the thermostat to maintain the correct temperature.

b. Stir the mud sample for 10 minutes with a high-speed mixer. Pour the mud sample into the mud cell, being careful not to fill the cell closer than 1.5 inches (38 millimeters) from the top to allow for expansion. Install the proper filter medium (see 5.3.1, Item b).

c. Complete the assembly of the cell, and with the top and bottom valves closed, place the mud cell in the heating jacket. Transfer the thermometer to the well in the mud cell.

d. Connect the high-pressure collection cell to the bottom valve, and lock in place.

e. Connect the regulated pressure source to the top valve and the collection cell, and lock in place.
f. With the top and bottom valves closed, apply the recommended back pressure (Table 2) for the test temperature to both top and bottom. Open the top valve, applying the same pressure to the mud while heating. Maintain this pressure until the test temperature is reached and stabilized.
g. When the temperature of the sample reaches the test temperature, increase the pressure on the top by 500 pounds per square inch (3450 kilopascals) over the back pressure being held, and open the bottom valve to begin filtration. Collect the filtrate for 30 minutes, holding the test temperature within ±5°F (±3°C) and maintaining the proper back pressure. If the back pressure should begin to rise, it can be reduced by cautiously drawing off a small portion of the filtrate. Time of heating of the sample in the filter cell should never exceed a total of one hour.
h. After the test period close both top and bottom valves on the pressure cell and bleed pressure from the regulators. Allow a minimum of 5 minutes for the filtrate to cool to avoid vaporizing, then cautiously drain and record the total volume. Also record the temperature, pressures, and time. Be sure to allow sufficient time for all the filtrate to drain from the receiver.

CAUTION: Pressure inside the filter cell will still be approximately 500 pounds per square inch (3450 kilopascals). Keep the cell upright and cool to room temperature before disassembly. Bleed pressure from cell before disassembling. Failure to do so could result in serious injury.

i. Remove the cell from the heating jacket, first making certain that the bottom and top valves are tightly shut and all pressure is off the regulators. Using extreme care to save the filter paper, place the cell upright, open the valve to bleed pressure from the cell contents and open the cell. Discard the mud, and retrieve the filter cake. Wash the filter cake on the paper with a gentle stream of water.
j. Measure and report the thickness of the filter cake to the nearest 1/32 inch (0.8 millimeter).

6 Water, Oil, and Solids

6.1 DESCRIPTION

The retort instrument provides a means for separating and measuring the volumes of water, oil, and solids contained in a sample of water-based mud. In the retort, a known volume of a whole water mud sample is heated to vaporize the liquid components which are then condensed and collected in a graduated receiver. Liquid volumes are determined directly from reading the oil and water phases in the receiver. The total volume of solids (suspended and dissolved) is obtained by the difference (total sample volume – liquid volume). Calculations are necessary to determine the volume of suspended solids since any dissolved solids will be retained in the retort. The relative volumes of low-gravity solids and weighting material can also be calculated. Knowledge of the solids concentration and composition is considered basic to viscosity and filtration control in water-based muds.

6.2 EQUIPMENT

The following equipment is used to separate and measure the volumes of water, oil, and solids.

a. Retort instrument (Figures 14 and 15):
Retorts of two sizes (10-cubic centimeter and 20-cubic centimeter) are commonly available. Specifications of these retorts are as follows:
1. Sample cup:

Item	Sample Cup Size	
Total volume	10-cm³	20-cm³
Precision	±0.05 cm³	±0.10 cm³

2. Liquid condenser: sufficient mass to cool the oil and water vapors below their vaporization temperature prior to leaving the condenser.
3. Heating element: sufficient wattage to raise the temperature of the sample above the vaporization point of the liquid components within 15 minutes without causing solids boil-over.
4. Temperature control: a temperature control is desirable. It should be capable of limiting the temperature of the retort to 930° ±70°F (500° ±20°C).

b. Liquid receiver:
1. Graduated cylinder, or tube.
2. Material: transparent and inert to oil, water, salt solution, and temperatures up to 90°F (32°C).
3. Specifications of graduation marks:

Precision	Graduate Size	
	10-cm³	20-cm³
	±0.10 cm³	±0.20 cm³
Graduation	0.10 cm³	0.20 cm³
Calibration method for marks	TC (to contain at 20°C)	
Scale to read	cm³ and/or volume percent	

c. Fine steel wool (in other words, No. 000).

Note: Liquid Steel Wool or similar products are not recommended for this application.

d. High-temperature silicone grease (used for a thread seal and a lubricant).
e. Pipe cleaners.

Recommended Practice Standard Procedure for Field Testing Oil-Based Drilling Fluids

API RECOMMENDED PRACTICE 13B-2
THIRD EDITION, FEBRUARY 1998

American Petroleum Institute

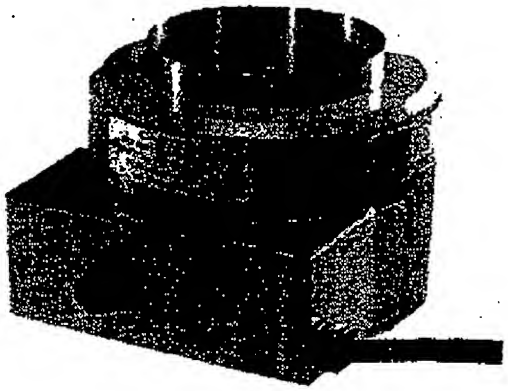


Figure 10—Heated Jacket With Cup

sample reaches the selected temperature. Record the temperature of the sample.

4.3.2.3 With the sleeve rotating at 600 rpm, wait for dial reading to reach a steady value (the time required is dependent on the mud characteristics). Record the dial reading for 600 rpm.

4.3.2.4 Shift to 300 rpm and wait for dial reading to reach steady value. Record the dial reading for 300 rpm.

4.3.2.5 Stir drilling fluid sample for 10 seconds at high speed.

4.3.2.6 Allow mud to stand undisturbed for 10 seconds. Slowly and steadily turn the hand-wheel in the direction to produce a positive dial reading. The maximum reading is the initial gel strength. For instruments having a 3-rpm speed, the maximum reading attained after rotation at 3 rpm is the initial gel strength. Record the initial gel strength (10 sec gel) in lb/100 ft² (Pa).

4.3.2.7 Restir the mud at high speed for 10 seconds and then allow the mud to stand undisturbed for 10 minutes. Repeat the measurements as in 4.3.2.6 and report the maximum reading as 10 minute gel in lb/100 ft² (Pa).

4.3.3 Calculation

$$\textit{Plastic Viscosity, cP} = \begin{bmatrix}\textit{600 rpm}\\ \textit{reading}\end{bmatrix} - \begin{bmatrix}\textit{300 rpm}\\ \textit{reading}\end{bmatrix} \quad (7)$$

$$\textit{Yield Point, lb/100 ft}^2 = \begin{bmatrix}\textit{300 rpm}\\ \textit{reading}\end{bmatrix} - \textit{Plastic Viscosity} \quad (8)$$

$$\textit{Apparent Viscosity, cP} = \frac{\textit{600 rpm reading}}{2} \quad (9)$$

Figure 11—Thermocup

5 Filtration

5.1 DESCRIPTION

5.1.1 Measurement of the filtration behavior and the filter cake characteristics of an oil-based drilling fluid are fundamental to the treatment and control of a drilling fluid, as are the characteristics of the filtrate, such as the oil, water or emulsion content.

5.1.2 Filtration characteristics of an oil-based drilling fluid are affected by the quantity, type and size of solid particles and emulsified water in the drilling fluid and by properties of the liquid phase. Interactions of these various components may be influenced by temperature and pressure.

5.1.3 Filtration tests are performed at both ambient (low) temperature and at high-temperature conditions to provide data for comparison purposes. Two filtration procedures are given—one procedure is for testing up to 350°F (177°C) and one procedure for testing from 351°F (177°C) up to 450°F (232°C). Use only the filtration equipment and procedure specified for the temperature required.

Note 1: No API low-temperature filtration test procedure for oil-based drilling fluids is specified herein, but can be performed much like the water-based drilling fluid test, found in API RP 13B-1.

Note 2: The API Low Temperature/Low Pressure filter press must have a filter area of 4520 to 4640 square millimeters, which is a diameter of 75.86 to 76.86 millimeters. The filter press gasket is the determining factor of the filter area. It is recommended that the filter press gasket used be tested by a conical gauge that has the maximum (76.86 millimeters) and the minimum (75.86 millimeters) marked on it. Any filter press gasket found out of these ranges (either larger or smaller than the markings) will be discarded.

5.1.4 Equipment for testing filtration up to and including 350°F (177°C) can be either the 175-, 250-, or 500-cm³ units. For testing above 351°F (178°C) only the 500-cm³ unit can be used and must be equipped with a thermocouple to measure the temperature of drilling fluid in the cell and also a porous stainless steel filter media must be used.

5.2 HIGH-TEMPERATURE/HIGH-PRESSURE TEST UP TO AND INCLUDING 350°F (177°C)

5.2.1 Equipment

Filtration testing equipment may not be rated to perform safely at temperatures and pressures called for in this procedure. It is essential to know and to follow the manufacturer's

recommendations regarding maximum operating temperature, pressure, and sample volumes. Failure to do so could result in injury.

a. The high-temperature/high-pressure filter press apparatus consists of:

1. A filter cell to contain working pressures up to 1300 psi (8970 kPa) at temperature.
2. A pressurized gas source, such as carbon dioxide or nitrogen with regulators.

Note: Nitrogen is preferred.

3. A heating system to heat to 350°F (177°C).
4. A high-pressure filtrate collection vessel maintained at proper back pressure (see Table 2) to avoid flashing or evaporation of the filtrate.

CAUTION: Do no use nitrogen oxide cartridges as pressure sources for high-temperature/high-pressure filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

Figures 12 and 13 are photographs of two filtration units used for testing up to 350°F (177°C).

b. Filter cell: the filter cell contains a thermometer well. It is fitted with a removable end, a filter-media support and with oil-resistant seals. Valve stems on each end of the cell can be opened or closed during the test.
c. Filter medium: Whatman No. 50 or S&S 576, or equivalent, filter paper.
d. Timer: to measure 30-minute interval. Mechanical or electronic.
e. Thermometer: to measure up to 500°F (260°C) with 5-inch (12.5-cm) or longer stem.
f. Receivers: 10-cm³ and 20-cm³ long, slender graduated cylinders, as used for oil, water and solids content. (See Section 6 and Figure 15).
g. Receiver (optional): 25-cm³ glass graduated cylinder (TC).
h. Field Mixer: cup type, to operate at 10,1000 to 15,000 rpm.
i. Ruler: to measure filter cake thickness in 1/32-inch increments.

5.2.2 Procedure—Temperatures to 350°F (177°C)

5.2.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 10°F (6°C) above the desired test temperature. Adjust the thermostat to the desired test temperature.

Note: If the filtration unit is equipped with a thermocouple to measure drilling fluid temperature inside the cell (test temperature) then that temperature should be monitored and maintained during the filtration test. Results may differ from this standard procedure, which uses the cell wall temperature as the test temperature. Under "Comments" section, note if the thermocouple method was used.

Table 2—Recommended Minimum Back Pressure

Test Temperature		Vapor Pressure of Water		Minimum Back Pressure	
°F	°C	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
350	177	135	932	160	1104

5.2.2.2 Stir the drilling fluid sample for 5 minutes using the field mixer. Pour the fluid sample into the filter cell, leaving at least 1 inch (2.5 cm) space in the cell, to allow for fluid expansion. Install the filter paper in the cell.

5.2.2.3 Complete the assembly of the filter cell. With upper and lower valve stems closed, place the cell in the heating jacket. Transfer the thermometer from the heating jacket into the well of the filter cell.

5.2.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place.

Note: Be sure the collection vessel is completely free of water or oil.

5.2.2.5 Connect the pressure-regulated gas source to the upper valve. Connect a similar gas source to the filtrate collection vessel and lock these connections in place.

5.2.2.6 While keeping the two valve stems closed, adjust the upper pressure regulator to a pressure 100 psi (690 kPa) higher than the "Minimum Back Pressure" value, as shown in Table 2. Next, set the lower regulator to the pressure "Minimum Back Pressure" shown in Table 2 for the test temperature. Maintain this pressure until the test temperature is reached.

Note: If time required to reach test temperature exceeds one hour, the heater may be defective and validity of the test is questionable.

5.2.2.7 When the sample reaches the selected test temperature, open the lower valve stem and immediately increase the pressure on the upper regulator to 500 psi (3450 kPa) higher than the back pressure. This will start the filtration process. Start the timer. Maintain the test temperature to within ±5°F (±3°C) during the test. If the back pressure rises above the selected back pressure during the test, cautiously draw off and collect a portion of the filtrate to reduce the back pressure.

5.2.2.8 Collect the filtrate in the long, slender graduated glass cylinder (or graduated cylinder). Read the 30-minute total (water plus oil) filtrate volumes. Also read volumes of solid and water phases, if present.

Note: A long, slender, glass cylinder, rather than a conventional graduated cylinder, is recommended because the long cylinder allows more accurate detection and measurements of volumes of oil, water and solids in the filtrate. Heating of the cylinder near an emulsion interface can improve separation of water, solids and oil in the filtrate.

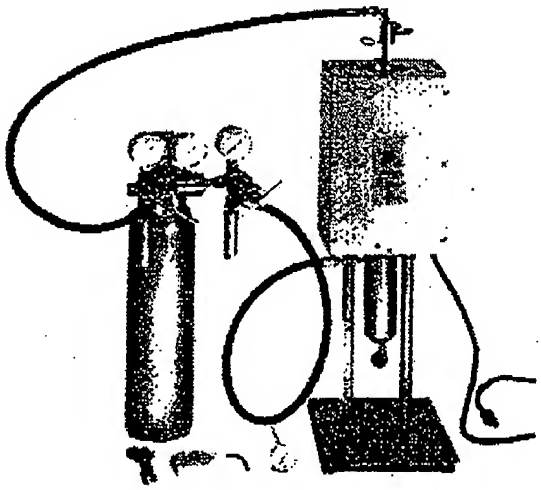


Figure 12—High Temperature Filter Press

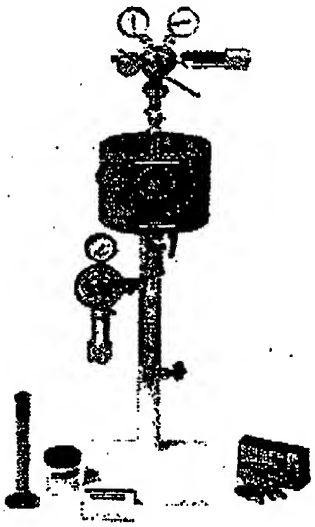


Figure 13—High Temperature Filter Press

5.2.2.9 Immediately after collecting the 30-minute filtrate, close the upper and lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the gas pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 125°F (52°C). Keep the cell upright during cooling, depressurization and disassembly.

CAUTION: Pressure in the filter cell can be high and dangerous, even after the cell is cooled. Be careful not to open the cell with pressure trapped inside.

5.2.2.10 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas exits stem. Carefully disassemble the cell. Be sure no pressure is trapped before dislodging the cap.

5.2.2.11 Pour the liquid from the cell.

5.2.2.12 Remove the filter cake on the paper. Measure the filter cake thickness at its center.

Note: Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics under "Comments" on the Oil Mud Report Form. To minimize settling, time for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

5.2.3 Calculation

5.2.3.1 Mathematically calculate the volume of filtrate for high-temperature/high-pressure filtration.

$$\textit{Filtrate vol., } cm^3 = 2\ (\textit{volume collected in 30 minutes, } cm^3) \quad (10)$$

Record the filtrate volume.

Note: Filtrate volumes (oil, water, solids) collected in the glass cylinder must be mathematically corrected to correspond to a standard filter area of 7.1 in^2 (4580 mm^2). High-pressure filter cells usually have half the standard filter area, 3.5 in^2 (2258 mm^2), thus all the observed volumes must be doubled before reporting.

5.2.3.2 Under "Comments" section of the report, record the doubled volumes of water or solids observed in the 30-minute filtrate.

5.2.3.3 Report the cake thickness to the nearest 1/32 inch (0.80 mm) on the Oil Mud Report Form.

5.3 HIGH-TEMPERATURE/HIGH-PRESSURE TEST 351°F (177°C) UP TO AND INCLUDING 450°F (232°C)

5.3.1 Equipment

CAUTION: Not all filtration testing equipment is rated to perform safely at temperatures and pressures called for in this procedure. It is essential to know and to follow the manufacturer's recommendations regarding maximum safe operating temperature, pressure and sample volumes. Failure to do so could result in injury. The 175- and 250-cm^3 filtration cells are not recommended for use at these higher temperatures and pressures.

a. The high-temperature/high-pressure filter press apparatus consists of:

1. A filter cell to contain working pressures up to 2250 psi (14,490 kPa) at a temperature of 450°F (232°C). The filter cell is equipped with an internal thermocouple to monitor temperature of a drilling fluid sample near its center in the cell. It is fitted with oil-resistant seals. Valve stems on each end of the cell can be opened or closed during a test.

2. A pressurized nitrogen gas source, with regulators.
3. A heating system to heat up to 450°F (232°C).
4. A high-pressure filtrate collection vessel maintained at proper back pressure to avoid flashing or evaporation of the filtrate. See Table 3.

CAUTION: Do not use nitrous oxide cartridges as pressure sources for high-temperature/high-pressure filtration. Under temperature and pressure, nitrous oxide can detonate in the presence of grease, oil or carbonaceous materials. Nitrous oxide cartridges are to be used only for Garrett Gas Train Carbonate Analysis.

b. Filter medium: Dynalloy® X-5, or equivalent, porous stainless steel disk to fit and seal in the cells.
c. Timer: to measure 30-minute intervals. Mechanical or electronic.
d. Thermometer: to measure up to 500°F (260°C) with 8-inch (20.3-cm) or longer stem.
e. Receivers: 10-cm³ and 20-cm³ long, slender graduated glass cylinders, as used for oil, water and solids content (see Section 6 and Figure 15).
f. Receiver (optional): 25-cm³ glass cylinder (TC).
g. Field Mixer: cup type, to operate at 10,000 to 15,000 rpm.
h. Ruler: to measure filter cake thickness in 1/32-inch (0.80 mm) increments.

Note: Dynalloy is a registered trademark of Memtec America Corporation.

5.3.2 Procedure—Temperatures from 351°F (177°C) to 450°F (232°C)

5.3.2.1 Place the thermometer in the well of the heating jacket. Preheat the jacket to approximately 10°F (6°C) above the desired test temperature. Adjust the thermostat to the test temperature.

5.3.2.2 Stir the drilling fluid sample for 5 minutes using the field mixer. Pour the fluid sample into the filter cell, leaving at least 1 inch (2.5 cm) space in the cell to allow for drilling fluid expansion. Install the porous stainless steel filter disk in the cell.

5.3.2.3 Complete the assembly of the filter cell. Install the thermocouple into the cell to monitor the temperature of the fluid near its center in the cell. With upper and lower valve stems closed, place the cell in the heating jacket. Connect the thermocouple to the temperature readout instrument and determine that it is reading correctly.

5.3.2.4 Connect the high-pressure filtrate collection vessel onto the lower valve stem and lock it in place.

Note: Be sure the filtrate collection vessel is free of residual water or oil.

5.3.2.5 Connect the pressure-regulated gas source to the upper valve. Connect a similar source to the lower collection vessel and lock these connections in place.

Table 3—Recommended Minimum Back Pressure

Test Temperature		Vapor Pressure of Water		Minimum Back Pressure	
°F	°C	psi	kPa	psi	kPa
212	100	14.7	101	100	690
250	121	30	207	100	690
300	149	67	462	100	690
350	177	135	932	160	1104
Limit of Normal Field Testing					
350	177	135	932	160	1104
400	204	247	1704	275	1898
450	232	422	2912	450	3105

5.3.2.6 While keeping the two valve stems closed, adjust the upper pressure regulator to a pressure 100 psi higher than the "Minimum Back Pressure" value, as shown in Table 3. Next, set the lower regulator to the pressure "Minimum Back Pressure" shown in Table 3 for the test temperature.

5.3.2.7 When the sample reaches the selected test temperature, as indicated by the thermocouple, open the lower valve stem and immediately increase the pressure on the upper regulator to 500 psi (3450 kPa) higher than the back pressure. This will start the filtration process.

Note: Start the filtration process immediately upon reaching test temperature, as read by the thermocouple in the fluid. This will minimize time during which solids settling is most likely to occur in the heated fluid.

5.3.2.8 Start the timer. Maintain the test temperature to within ±5°F (±3°C) during the test. If the back pressure rises above the selected back pressure during the test, cautiously draw off an collect a portion of the filtrate to reduce the back pressure.

5.3.2.9 Collect the filtrate in the long, slender glass cylinder, or the graduated cylinder. Read the 30-minute total (water plus oil) filtrate volume. Also read volumes of solid and water phases, if present.

5.3.2.10 Immediately after collecting the 30-minute filtrate, close the upper and lower valve stems to contain the pressure. Following the manufacturer's detailed instructions, bleed pressure off the regulators and hoses, then disconnect the gas pressurization system. Remove the cell from the heating jacket and allow cell to cool to below 125°F (52°C). Keep the cell upright during cooling, depressurization, and disassembly.

CAUTION: Pressure in the filter cell can be high and dangerous, even after the cell is cooled. Be careful not to open the cell with pressure trapped inside.

5.3.2.11 Bleed pressure from the filter cell by slowly opening the upper valve stem. Avoid spraying drilling fluid as gas

exits stem. Carefully disassemble the cell. Be sure no pressure is trapped before dislodging the cap.

5.3.2.12 Pour the liquid from the cell.

5.3.2.13 Remove the filter cake on the Dynalloy disk. Measure the filter cake thickness at its center.

Note: Settling of solids onto the filter cake may have occurred during the test. Observe indications of this, such as an abnormally thick cake or coarse texture. Record these cake characteristics under "Comments" on the Oil Mud Report Form. To minimize settling, time for heat-up and cool-down should be minimized and the cake should be recovered and examined promptly.

5.3.3 Calculation

5.3.3.1 Mathematically calculate the volume of filtrate for high-temperature/high-pressure filtration.

$$\text{Filtrate vol., cm}^3 = 2\ (\text{volume collected in 30 minutes, cm}^3) \quad (11)$$

Record the filtrate volume on the Oil Mud Report Form.

Note: Filtrate volumes (oil, water, solids) collected in the glass cylinder must be mathematically "corrected" to correspond to a standard filter area of 7.1 in² (4580 mm²). High-pressure filter cells usually have half the standard filter area, 3.5 in² (2258 mm²), thus all the observed volumes must be doubled before reporting.

5.3.3.2 Under "Comments" section of the report, record the doubled volumes of water or solids observed in the 30-minute filtrate.

5.3.3.3 Report the cake thickness to the nearest 1/32 inch (0.80 mm) on the Oil Mud Report Form.

6 Oil, Water, and Solids Content

6.1 DESCRIPTION

6.1.1 A retort test measures water and oil released from an oil mud sample when heated in a calibrated and properly operating "Retort" instrument.

6.1.2 Knowledge of water, oil and solids content is fundamental to proper control of mud properties such as: oil/water ratio, rheology, density, filtration and salinity of aqueous phase. Knowledge of solids in an oil mud is essential to evaluation of solids control equipment.

6.1.3 In a retort test, a known volume of oil mud is heated in a retort instrument to vaporize the liquid components. These vapors are then condensed and collected in a precision graduated receiver. Volume percent retort solids is calculated by subtracting the total liquid volume from the starting volume of oil mud.

6.2 EQUIPMENT

Following is a list of equipment needed:

a. Retort instrument: The recommended oil mud retort instruments are 10-cm³ or 20-cm³ volume with an external heating jacket (see Figure 14).

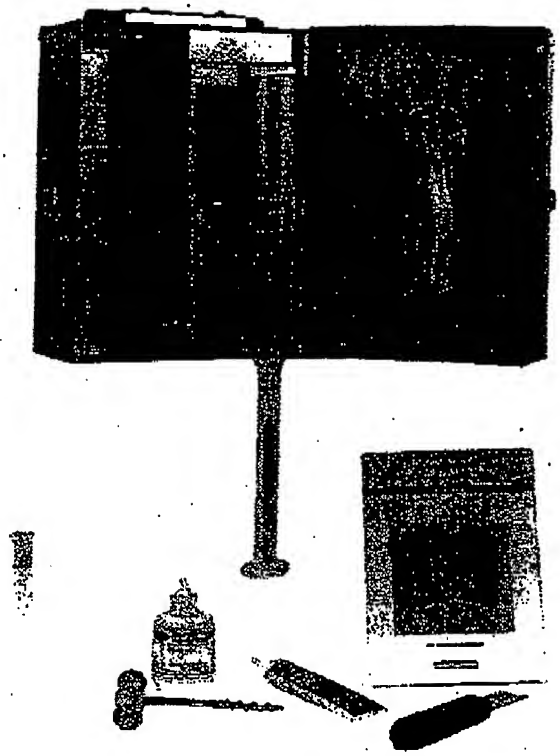


Figure 14—Retort for Liquid and Solid Content

Note: A 50-cm³ retort, although not specified, is available.

Specifications for retort instruments are as follows:

1. Retort assembly: retort body, cup and lid.
   (a) Material: 303 stainless steel, or equivalent.
   (b) Volume: retort cup with lid.

Cup volume:	10-cm³	20-cm³
Precision:	±0.05 cm³	±0.1 cm³

Note: *Verification of retort cup volume.* The retort cup volume with lid should be verified gravimetrically. The procedure and calculations are in 6.5.

2. Condenser: capable of cooling the oil and water vapors below their liquification temperature.
3. Heating jacket: nominal 350 watts.
4. Temperature controller: capable of limiting temperature of retort to 930 ±70°F (500 ±38°C).

b. Liquid receiver: specially designed cylindrical glassware with a rounded bottom to facilitate cleaning and a funnel-shaped top to catch falling drops (see Figure 15).

Specifications for liquid receivers are the following:

Total volume:	10-cm³	20-cm³
Precision (0 to 100%):	±0.05 cm³	±0.05 cm³
Outside diameter:	10 mm	13 mm
Wall thickness:	1.5 ± 0.1 mm	1.2 ±0.1 mm